Fabricating Sub-collimating Grids for an X-ray Solar imaging Spectrometer Using LIGA Techniques

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ABSTRACT

We are fabricating sub-collimating X-ray grids that are to be used in an instrument for the High Energy Solar Spectroscopic Imager (HESSI), a proposed NASA mission. The HESSI instrument consists of twelve rotating pairs of high aspect ratio, high Z grids, each pair of which is separated by 1.7 meters and backed by a single Ge detector. Tbe pitch for these grid pairs ranges from 34 µm to317 µm with the grid slit openings being 60% of the pitch. For maximum grid X-ray absorbing with minimum loss of the solar image, the grid thickness-to-grid-s]it ratio must be approximately 50:1, resulting in grid thicknesses of 1 to 10 millimeters. For our proof-of-concept "grids we are implementing a design in which a 34 µm pitch, free-standing *PMMA* grid is fabricate.d with 20 µm wide slits and an 800 µm thickness. Stiffeners that run perpendicular to the grid are placed every 500 µm. After exposure and developing, metal, ideally gold, is electrodeposited into the free-standing PMMA grid slits. The PMMA is *not* removed and the metal in tbe slits acts as the X-ray absorber grid while the PMMA holds the individual metal pieces in place, the PMMA being nearly transparent to the X-rays coming from the sun. For optimum imaging performance, the root-mean-square pitch of the two grids of each pair must match to within 1 part in 10000 and simultaneous exposures of stacked sheets of PMMA have insured that this requirement is met.

Keywords: x-ray collimators, x-ray grids, thick films, high aspect ratio structures, MEMS electroplating, LIGA, x-ray lithography, deep etch x-ray lithography

BRENNEN, R.

2. INTRODUCTION

Thick grids, filters, and other repeated pattern structures have been fabricated by high aspect ratio means for some time[1]. These structures have various requirements governing the minimum required thickness, including strength, structural stability, and optical collimation. Thick, fine-featured grids are required for the High Energy Solar Spectroscopic Imager (1111 SS1)[2], a proposed NASA mission that will promote the understanding of solar particle acceleration and explosive energy release in the magnetized plasmas at tbc Sun. These grids have a required minimum pitch that is determined by the. expected angular resolution of the imager and have a required minimum, and maximum, thickness that is governed by the photon energy bandwidth desired for the X-ray imager.

The HESSI mission proposes to perform high resolution imaging and spectroscopy observations in the soft X-ray, hard X-ray, and gamma-ray regimes, with finer angular resolution (nearly 2 arcseconds) and finer energy resolution (approximately 1 keV) than has been previously possible[2]. This combination of imaging and spectroscopy is achieved with a set of Rotating Modulation Collimators placed in front of an array of cooled germanium and silicon detectors. A set of 12 hi-grid collimators, each of which consists of a pair of identically pitched, widely-separated grids, is used to provide the imaging. Each grid consists of a planar array of equally-spaced, parallel, X-ray opaque, slats separated by X-ray transparent slits. If the slits' of each grid arc parallel to each other, and the pitch is identical for the two grids, [hen the transmission through the grid pair depends on the direction of incidence of the incoming X-rays as shown in Fig. 1. For slits and slats of equal width, tile transmission varies between zero and 50% depending on whether the shadows of the slats in the top grid fall on the slits or slats of the lower grid. A complete transmission cycle from zero to 50% and back to zero corresponds to a change in source direction that is given by p/L, where L is the separation bet ween the grids. The angular resolution is then given by $p/(2L)$. Rotation of a grid pair assembly provides the change in source direction and by using the 12 differently pitched grid pairs, a two dimensional image of the source can be reconstructed from the Fourier components determined from the modulated signals.

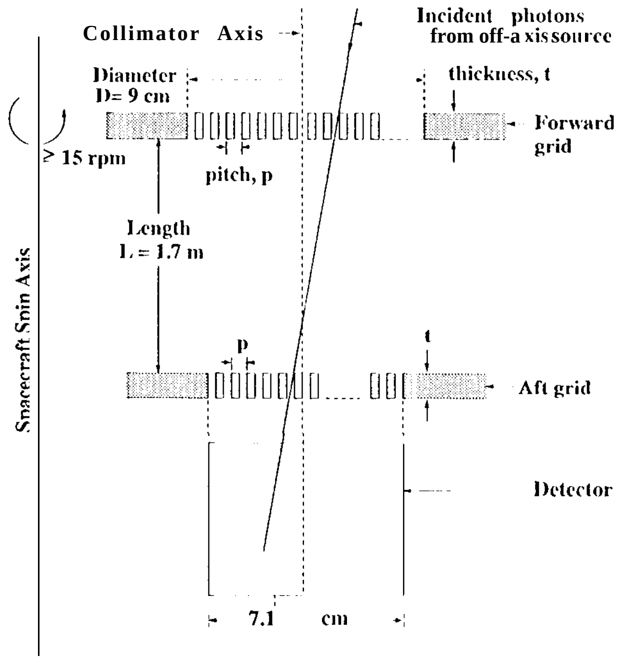

Figure 1 **Schematic of forward and aft grids in the Rotation Modulation Collimator. Note off-axis photon path.**

BRENNEN, R.

3. GRID REQUIREMENTS AND DESIGN

3.0. Grid requirements

The finest angular resolution is a function of the smallest pitch of the grids. For a minimum resolution of 2 arcseconds and a grid separation of 1.7 meters, the pitch of the finest grid must be 34 µm. Each grid must be almost perfectly identical ; the RMS pitch of tbc grids in each pair must correspond to within 1 part in 10000. The tolerances for each grid as to slat width, thickness variation, and slat position are much less stringent, being on the order of 5% to 1 (Y%. The active grid area of the lower, or aft, grid must have a diameter of 7.1 centimeters and the front grid a diameter of 8.9 centimeters.

The thickness of the grids is determined by two factors. For contrast at the high photon energies, the grids must be fabricated from a high-Z material and be as thick as possible for maximum photon absorption, However, the field of view of tbc top grid through each slit must be at least 1 degree for full-Sun plus flare imaging. 'l'his requires that the aspect ratio of tbc slit be at most 57:1. The optimum grid slit width is 20 µm for the 34 µm pitch grid, resulting in a maximum thickness for this grid of 1.1 millimeters. This thickness allows collimation of photons with energies of less than 300 keV.

The pitch of the coarsest grid will be 2 millimeters with a corresponding thickness of 2.5 centimeters, allowing collimation of photons of up to 2(I MeV. Techniques such as electrodischarge machining (EDM) and tungsten foil stacking using spacers can be used to fabricate grids with pitches from 2 millimeters down to approximately 100 pm. However, tbc 10^{-4} correspondence for the RMS pitch for any grid pair has not yet been met by the above methods for grids with pitches below] 00 µm. The LIGA processing technique provides a unique ant] ideal method for fabricating the three required grids with pitches smaller than 100 µm.

3.1. Grid design

The original design for the 34 µm pitch grids had a series of parallel slats stiffened every 1000 µm by a slat that was oriented perpendicular to the fine parallel slats. This design was modified when it was determined that the resist mold for this configuration would require 1000 µm tall features with a footprint of only 20 by 1000 µm as shown in Fig. 2. The second configuration had larger footprint as is also shown in Fig. 2. However, while the footprint was larger, the long times required for developing down 1 mm meant that some parts of the poly methyl methacrylate (PMMA)/substrate interface would continue to fail.

A modification to this developing method was introduced by performing the exposures using free-standing sheets of PMMA as shown in Fig. 3. The PMMA triple cell grid sample was partially developed, down to a depth of about 30 to 60 µm anti then a 2000 Å metallic seed layer was Sputtered over and into the resulting features. A 25. µm metallic layer- was then electroplated into the features using the sputtered metal as a plating strike. The sample was then completely developed down through the exposed resist fi om the unplated side to the side covered with the plated metal. However, the 800 µm tall mo]d pieces were not stiff enough to maintain the required registration in the grid slats at the top of the mold and the samples plated using these molds did not have acceptable registration at the top.

A completely different grid design is now used to fabricate the grids. This design consists of a very rigid mold structure which does not require a substrate. A free-standing sheet of PMMA that is not mounted on a substrate is exposed such that, after developing, the mold structure is a free-standing single cell PMMA grid mold as shown in Fig. 4. The open slits in this grid are filled with electroplated metal which acts as the X-ray absorbing collimating grid since the PMMA slats between the metal-filled slits are acceptably transparent to high energy X-rays down to approximately 10 keV photons. This grid can collimate X-rays from 10 keV to 300 keV, meeting the specifications for tbc 34 µm pitch grid, For lower energy X-rays, a grid must be used that has no material between the absorbing slats[3].

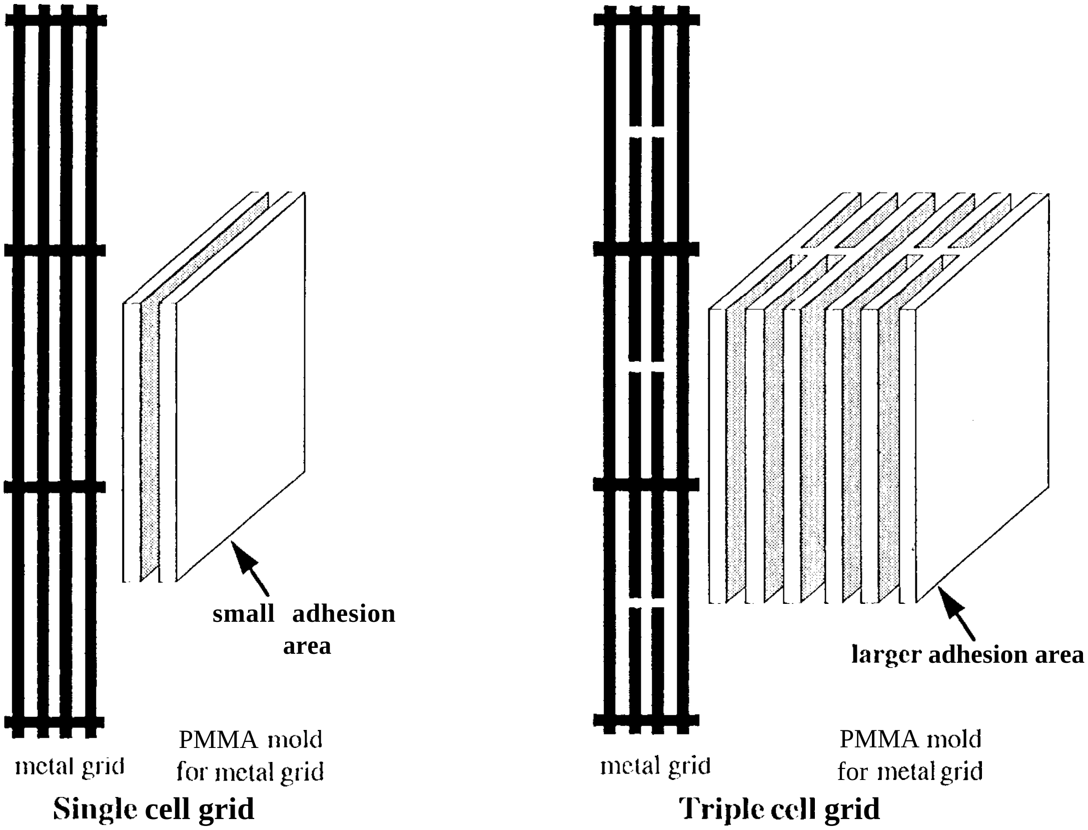

Figure 2. The footprint of the original PMMA mold features for the single cell grid did not provide sufficient adhesion and the mold fell apart. The triple cell grid mold had a much greater adhesion area.

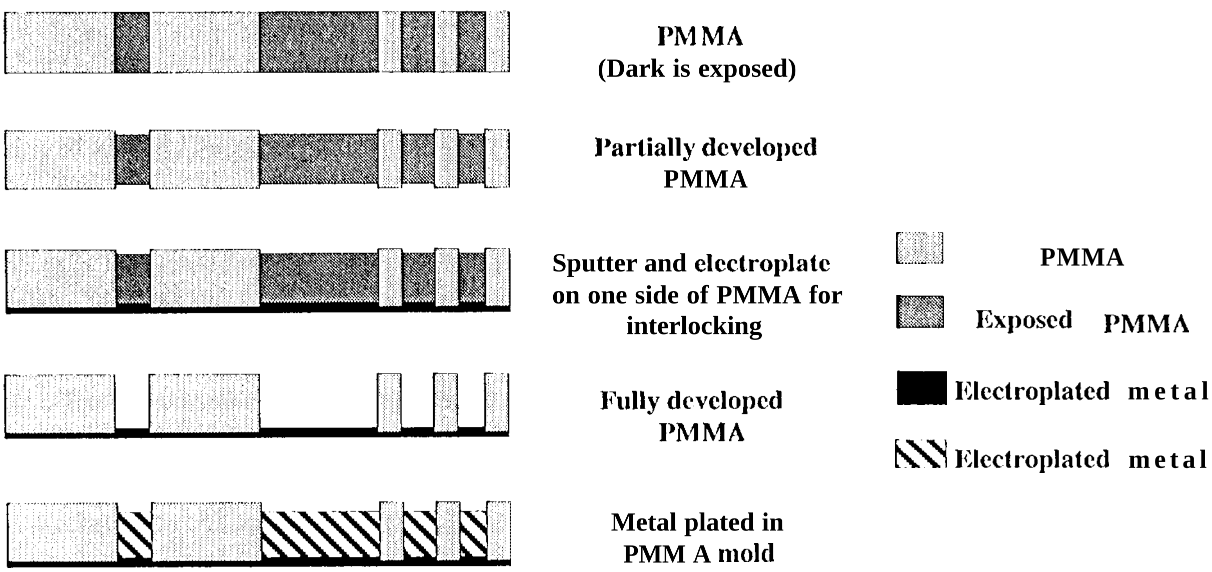

Figure 3. Alternative method of developing and plating to increase resist adhesion during plating.

An oxygen plasma cleaning is performed on the resist side of the wafer. This step is required to clean the surface of the gold seed layer. Without this step, the electroplating process is not reliable anti large variations in plating rate occur over the wafer. In extreme cases, no plating occurs at all. Initial efforts in electroplating gold into the resist were made using a potassium gold cyanide bath. This basis bath is not compatible with the AZP4620 resist and delamination of the resist from the substrate is only eliminated by doing an initial plating of copper. An acidic gold electrolyte has been found anti the electroplating of gold is now performed using a Technics 25E sodium gold sulfite bath with a pH of 3.8 and a temperature of 51.6 °C. The current density is 0.003 A/cm^2 (3 A/ft^2). The gold is plated up to the top of the resist. The photoresist is then removed and the mask is mounted in an aluminum holder plate.

4.2 Exposure of the PMMA

4.21 Exposure light source

Exposures of the PMMA were made on two synchrotrons -- the Advanced Light Source (ALS) at Lawrence Berkeley Laboratory in Berkeley, California and the Stanford Synchrotrons Radiation Laboratory (SSRL) in Palo Alto, California. The ALS bending magnet photon output has a characteristic energy of 1.6 keV when running at 1.5 GeV and a characteristic energy of 3.1 keV when running at i.9 GeV. Beamline 10.3.2 at the ALS has an acceptance of 2 milliradians and a beamline length of 30 meters resulting in a horizontal exposure width of 6 centimeters. SSRL has a characteristic energy of 4.7 keV with an 11 centimeter exposure spanning 5.5 milliradians. Because the X-ray masks for the grids are currently fabricated only on 200 μm thick silicon substrates, grid exposures are carried out only at SSRL and at the ALS during 1.9 GeV operation since an insufficient flux of energy from the 1.5 GeV output can pass through the silicon mask substrate

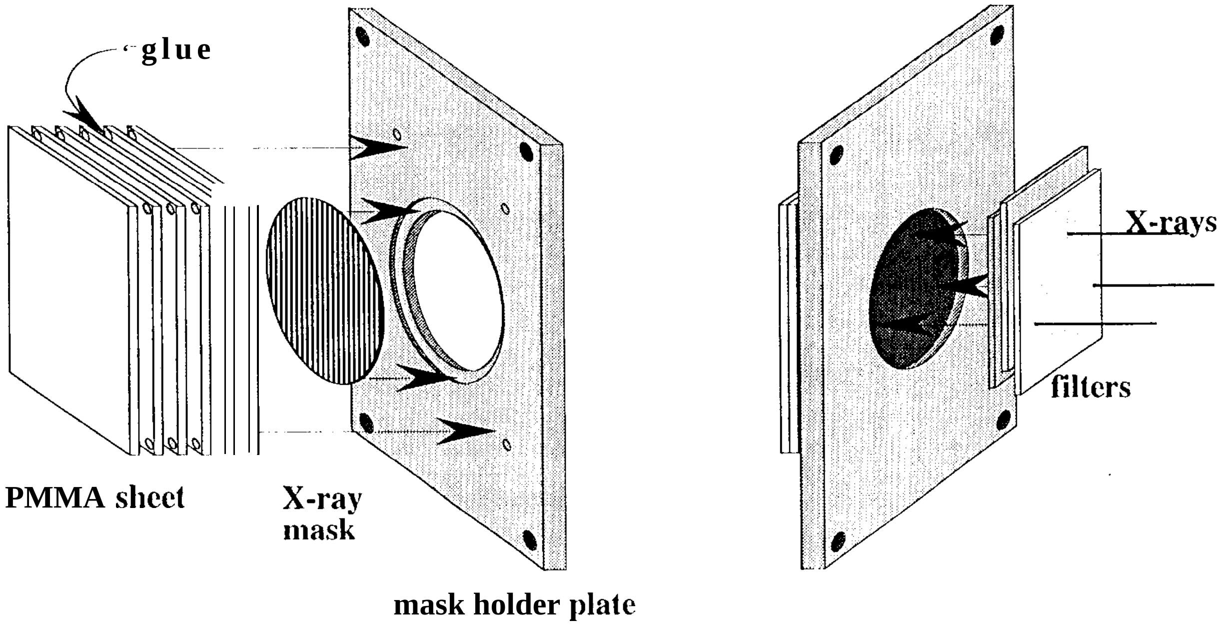

Figure 5. Assembly schematic showing the X-ray mask, its holder, and multiple sheets of PMMA on left and the X-ray exposure direction on the right.

4.2.2 Sample preparation

The exposures at both the ALS at 1.9 GeV and at SSRL at 3.0 GeV are made using stacks of PMMA sheet, AtoHaas Plexiglas® G with a molecular weight of between 1 and 1.5 million. Although the target thickness of the final 34 pm pitch grids is 1.1 millimeter, 800 μm thick sheets are used for each layer since they meet the minimum thickness requirements. Each 9 centimeter square of PMMA is annealed at 100 degrees Centigrade for three hours and then cooled at ().25 °C per minute to room temperature. Typically, six sheets ate glued together at the corners using a cyanoacrylate adhesive, as shown in Fig. 5. This stack is then glued directly to the mask holder plate with a separation of approximate] y 200 to 500 μm between the mask and top sheet of I'M MA. Previous methods of holding the mask and PMMA sheets in place with respect to one another relied upon clamping mechanisms but it was determined that the gluing ensured continued alignment. It should be noted that tbc alignment of mask feature to substrate feature prior to exposure has not yet been necessary.

4.2.3 Exposures

The exposures at SSRL were set such that grid slats defined on the mask ran horizontally. In this manner, the maximum angle of the sidewalls, *due to beam angle,* can be assumed to be 0.5 μm per 1 millimeter height since the angular spread of the X-ray beam in the vertical direction is 0.5 milliradians. Further, since any horizontal pattern edge passes both the top and the bottom of the X-ray beam and the highest energy photons are concentrated vertically in the central portion of the exposure area, tbc exposed section of the resist can be considered to be better than the above 0.5 milliradians.

The target exposure dose is between 2 kJ/cm^3 and 20 kJ/cm^3. Since the PMMA absorbs the lower energy photons more readily than high energy ones which can penetrate more deeply into the resist, the front or top of the I'MMA stack receives a higher dose than the back. In order that all the exposed I'MMA receive a dose high enough to be developed while not being overdosed, the ratio between the front and back doses should remain below about 5. The filters through which the synchrotrons X-ray beam passes before exposing the PMMA resist include beryllium, aluminum, the silicon substrate of the X-ray mask, and for some exposures, Kapton®. The target dose for the back side of the last I'MMA sheet is typically 2.5 kJ/cm^3 resulting in maximum doses on the front of approximately 7 kJ/cm^3 for exposures at SSRL and 10 kJ/cm^3 for exposures at the ALS with exposure times of 4 hours and 13 hours, respectively, pcr 1 cm of vertical scan. No cooling is currently used for the mask or substrate. The samples arc scanned in front of the beam at speeds ranging from 35 to 70 mm/s.

4.3 Developing of exposed resist

Developing of the exposed resist is performed using the "GG" developers - 60% 2-(2 -butoxyethoxy) ethanol, 20% morpholine, 15% water, 5% ethanolamine. The samples arc developed at 35°C in 1000 ml of the developer using a magnetic stirrer for agitation. They arc then rinsed in 1000 ml of 35°C "GG" rinse, 80% 2-(2-butoxyethoxy) ethanol, 20% water, and then rinsed again in 35°C DI water.

Since the exposed samples of resist are free-standing sheets of PMM A and the developed pattern is a self-supporting grid, they can be developed from both sides of the sheet simultaneously. 'l'he 800 μm thickness of the sheets, in addition 10 the unexposed surrounding area, ensures a structurally sound geometry that remains intact through the developing process. The samples are immersed in the developer in a such that the flow over the surface of the resist is parallel to the lines of the grid. Samples in which the flow is perpendicular to the grid develop very slowly, Full development times for parallel flow developing range for an 800 μm thick grid range from 1 hour to 12 hours depending on the exposure dose. These long develop times result from the small size of the slits which arc nominally 17 μm wide. After the sample develops through, the sample is turned for 10 to 15 minutes such that the 11ow is perpendicular to the *surface* of the sample. This allows rapid direct passage of the developer through the grid slits, The subsequent rinses arc both performed with this perpendicular orientation of the sample, the first rinse for 30 to 60 minutes and the DI water rinse for at least 1 hour. Long rinse times in the DI water rinse reduce the likelihood of the residue which can remain after developing.

The exposed samples of the original design were developed from one side only. Stirring agitation was used to develop these samples and duc to the extremely small size of the exposed areas, developing took up to 3 days to perform. The unexposed areas of these samples were not attacked despite the long develop time. It was very important to remove the developer with a high concentration of dissolved PMMA from within the deep slots and this was performed by using these long develop times. Although ultrasonic agitation increased transport greatly, the fine geometry of the grid molds could not withstand this type of agitation and the mold was destroyed when it was used.

It must be determined whether or not the PMMA grid has been fully developed and all the slots are completely cleared of undeveloped exposed resist and/or residues. One method that we have used with good results has been an evaporation of aluminum through the grid slits. If the slits are developed through, the evaporated aluminum can pass through. The developed sample is placed over a glass plate and then the glass plate and sample arc mounted on a rotating chuck in an evaporator. The rotating chuck ensures that the evaporating aluminum, traveling line-of-sight, can pass through the slits if they are free of obstruction. After the evaporation, the aluminum pattern on the glass plate is examined to determine the quality of the developing.

4.4 Electroplating

All electroplating for the HESSI grids is performed using a copper plating bath. Once this process is fully stable, the grids will be plated using a gold batb which has electroplating characteristics similar to that of copper baths.

Tbc developed I'MMA grid mold is mounted on an copper plate with a very clean surface. The mold is attached to the plate using a highly conforming, non-conducting tape such that only the grid portion of the sample is open to the electroplating bath. The sample is immersed in a commercially available acid copper plating bath (Sel-Rex Cubath M). Several types of bath agitation have been tested including mechanical agitation, bubbling agitation, simple flow over the surface (not laminar flow). Currently, only enough agitation is used to ensure a good concentration of electrolyte at the top surface of the grid pattern since diffusion appears to be the rate-limiting mechanism in filling the grid mold while still maintaining a constant plating rate over the whole area of the pattern. This diffusion is comparatively slow and the plating up of 800 μm of copper takes several days.

S. RESULTS ANI) CONCLUSION

Many X-ray masks for HESSI grid patterns have been successfully fabricated and used in exposures with synchrotron X-ray radiation sources. The absorber thickness on the masks ranged from 20 to 27 pm. For exposures at SSRL, 25 pm thick gold absorber is acceptable but should ideally be closer to 40 μm for optimum contrast between exposed and unexposed areas and therefore better geometric definition in the developed part.

The stacked PMMA sheet configuration ensures that the registration of the grid, the distance between the slat centerlines, is the same from sheet to sheet. The size of the features will vary for the. six sheets since diffraction and photoelectron production effects will be different for each sheet but theory shows that this effect will be small - on the order of 0.5 μm horizontal feature dimension deviation per 1 mm thickness of resist.[6]

One factor that can I-educe the accuracy of the grid registration is heating of the mask and/or the PMMA sheets. Some of the samples with large exposure areas have uneven developing over the area of the exposure. Some areas have all the PMMA removed, not leaving even the nominally unexposed areas. These areas are at the top and bottom of the scanned area on the resist. This variability is not due to exposure *through* the absorber since the masks usually have an absorber thickness constant to within 10% over the whole. wafer. Therefore this variability y is probably duc to the heat generated by the beam acting on the mask and causing it to expand vertically up *and* down repeatedly such that nominally unexposed areas are exposed due to repeated mask/resist misalignment. Another possibility is resist heating caused by preferential heating of the ends of the scan area due to the shorter period of time between exposure of the PMMA at the ends compared to the middle of the scan area which is scanned evenly every one half of one scan period. The largest exposed samples have 34 μm pitch grid patterns 6 cm in diameter but the largest successfully exposed *and* developed I'MMA grid mold samples arc 1.5 inches square.

Tbc exposures include both the original and current designs for the grids. The fabrication method using post-exposure application of a sputtered/plated electroplating surface does not solve the grid registration problem during electroplating of the original design. however, it is shown that the method does allow a pattern to be exposed in a free-standins sheet of I'MMA and be attached to a substrate so that free-standing features of the PMMA arc not removed during the subsequent developing and electroplating processes, as pictured in Fig. 6.

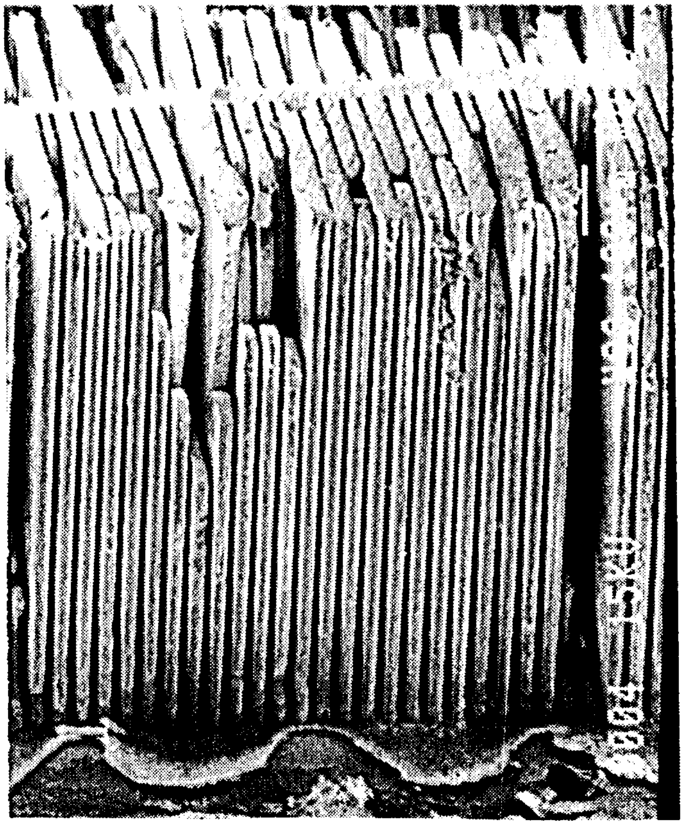

Figure 6. SEM of electroplated copper with original design PMMA grid mold removed. The top has been planarized with a rough grit abrasive. Note the joint about 30 μm from the substrate at the bottom of each blade where they meet the prc-develop, post-exposure sputtered/plated backing. The rough plating base surface is due to electrolyte that seeps behind the free-standing mold during the plating process.

The second method, in which the PMMA sheet is exposed, developed, and then attached to a conducting plate, is successful in allowing structures to be plated, although individual free-standing features of PMMA were not attempted. Results of this process arc shown in Figs. 7 – 9. These parts have not been planarized so that the plating characteristics can be seen more easily. These sample grids, actually the reverse of stiffened grids, are approximately 400 μm thick (tall) and show moderate plating uniformity except for a few features which plated much faster than the rest. This is probably due to an electric field concentration at the entrance to the slit delineating the features, since tile features that plated faster are usually smaller than the rest of the fcatures[7].

The current issues to be examined for this project include the uneven exposure and development of large exposed areas, uneven electroplating in the PMMA grid mold, and methods for increasing the development rate. The energy absorbed by the silicon and gold mask is on the order of 1 watt per horizontal centimeter at SSRL, totally 6 watts for a 3 inch wafer mask. This can be reduced by either cooling the mask or by using a pre-filter and a mask substrate that is more X-ray transparent than the current 200 μm of silicon.

BRENNEN, R

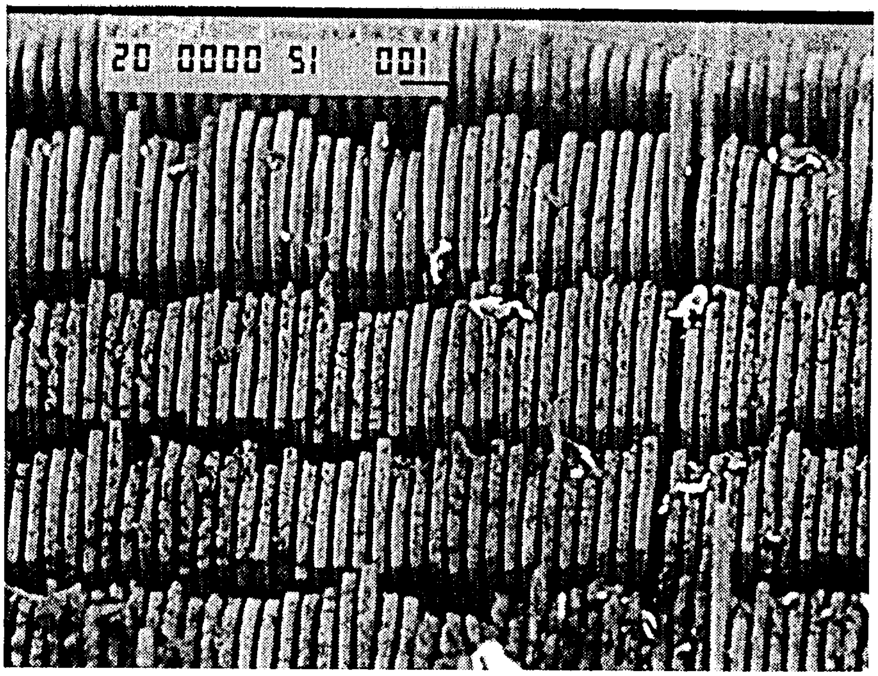

Figure 7. **SEM of electroplated copper with PMMA grid mold removed. Samples are 400 μm tall and on a 34 μm pitch. Note that the electroplating has not been planarized. The slightly wider slit to the right is an artifact from the mask.**

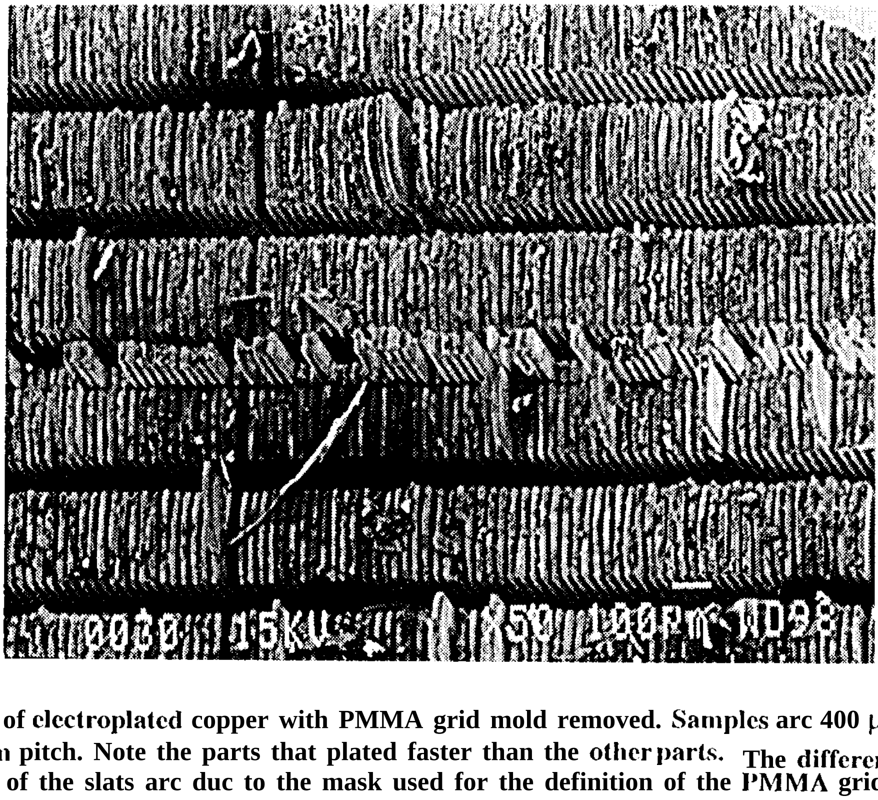

Figure 8. **SEM of electroplated copper with PMMA grid mold removed. Samples are 400 μm tall and on a 34 μm pitch. Note the parts that plated faster than the other parts. The differences in length of some of the slats are due to the mask used for the definition of the PMMA grid stiffeners. The slightly wider slit to the left is an artifact from the mask.**

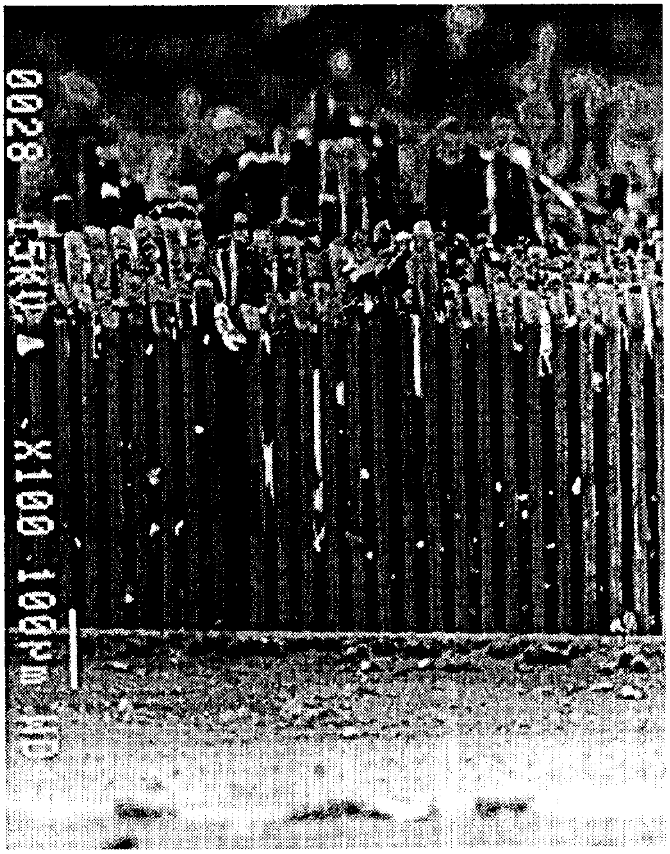

Figure 9. **SEM of electroplated copper with PMMA grid mold removed. Samples are 400 µm tall and on a 34 µm pitch. The slightly wider slit to the left is doe to the mask design. The rough plating base surface is due to electrolyte that seeps behind the free-standing mold during the plating process. There is some residue between the individual pieces of copper due to the incomplete removal of the PMMA mold.**

6. ACKNOWLEDGMENTS

The research described in this paper was performed by the Center for Space Microelectronics Technology, Jet Propulsion Laboratory, California Institute of Technology, and by Sandia National Laboratory, Livermore, and was sponsored by the National Aeronautics and Space Administration, Office of Space Access and Technology. Synchrotrons X-ray beam time was provided by the Advanced Light Source at Lawrence Berkeley Laboratory and by the Stanford Synchrotron Radiation laboratory at the Stanford linear Accelerator Center.

7. REFERENCES

1. Becker, E. W., W. Ehrfeld, P. Hagmann, A. Maner, and D. Munchmeyer, "Fabrication of microstructures with high aspect ratios and great structural heights by synchrotron radiation lithography, galvanoforming, and plastic moulding," Microelectronic Engineering, vol. 4, pp. 35-36, 1986.

2. Lin, R. P., Principle Investigator, "The High Energy Solar Spectroscopic Imager (HESSI)", a proposal submitted to **NASA in** response to the MIDEX Announcement of Opportunity, June 21, 1995.

3. Brennen, R. A., Hecht, M.H., Wiberg, D. V., Manion, S. J., Bonivert, W. D., Hruby, J. , Kruglick, E., Pister, K. S. J., "Fine pitch grids for an x-ray solar imaging spectrometer fabricated by optical lithography and XeF_2 etching," to be presented at Micromachining and Microfabrication '95, SPIE, Austin, Oct. 23-24, 1995.

4. Mohr, J., Ehrfeld, W., Munchmeyer, D., Stutz, A., "Resist technology for deep-etch synchrotrons radiation lithography," Makromolekulare Chemie, Macromolecular Symposia Series, V. 24, pp. 231-251, 1989.

5. Guckel, H., T.R. Christenson, K.J. Skrobis, D.D. Denton, B. Choi, E.G. Lovell, J.W. Lee, S.S. Bajikar, and T.W. Chapman, "Deep X-ray and UV lithographies for micromechanics," Technical Digest, IEEE Solid-State Sensor and Actuator Workshop, pp. 118-122, June 4-7, 1990.

6. Barrington, R.O., "1.1(3A technology, " Proceedings Prospector V Workshop, Park City, Utah, March 21-24, 1993.

7. Bade, K., A. Thommes, K. Leyendecker, W. Bacher, "Effects of electroplating at high aspect ratio micropatterned electrodes," presented at High Aspect Ratio Microstructure Technology, Karlsruhe, July 3-5, 1995.

BRENNEN, R